भारतीय मानक
Indian Standard

IS 12795 : 2020

रैखिक ऐल्किल बेंजीन — विशिष्टि

(पहला पुनरीक्षण)

Linear Alkyl Benzene — Specification

(First Revision)

ICS 71.100.40

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Soaps and other Surface Active Agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1989. Linear alkyl benzene (LAB) is one of the major raw materials used for manufacturing surfactant for use in synthetic detergents. It is a biodegradable material and is therefore finding greater acceptability since it has been found that effluent water containing detergents based on non-biodegradable raw materials could not be rendered harmless even after rigorous water treatment.

Linear alkyl benzene is an important major raw material for manufacture of surfactant for use in household and industrial synthetic detergent powders, tablets and liquid detergents. The limitations of soaps for use in hard water areas have led to the development of synthetic detergents industry. The shortage of oils and fats, the main raw materials for the production of soaps and increase in their demand for edible purposes has further helped the growth of synthetic detergent industry in India.

In the formulation of this standard, considerable assistance has been derived from Indian Petrochemicals Corporation Limited and Universal Oil Products, USA.

This standard was originally published in 1989. In this revision, the requirements of linear alkyl benzene content, refractive index and tetralins have been incorporated. Requirement of paraffin, ready biodegradibility of sulphonate and chain length distribution have been modified. Bromine number has been replaced by Bromine index. Test methods have been updated. Further, amendments issued to the previous version of the standard have been incorporated after review.

This standard was prepared by CHD 25 Soaps and other Surface Active Agents technical committee. The list of experts who made significant contribution to the revision of this standard is given at Annex J.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LINEAR ALKYL BENZENE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for linear alkyl benzene.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No./International Standard	*Title*
286 : 2018	Methods of sampling and test for soaps (*third revision*)
1070 : 1992	Reagent grade water — specification (*third revision*)
1447 (Part 1) : 2000	Petroleum and its products — Methods of sampling: Part 1 Manual sampling (*first revision*)
1448	Method of test for petroleum and its products
(Part 13) : 1960	Colour by Lovibond tintometer
(Part 14) : 2019	Colour by Saybolt Chromometer (*first revision*)
(Part 18) : 1991	Distillation of petroleum products (*second revision*)
(Part 19) : 2015	Detection of thiols and other sulphur species — Doctor test (*second revision*)
(Part 21) : 2019/ ISO 2719:2016	Determination of Flash Point — Pensky-Martens Closed Cup Method (*fourth revision*)
(Part 32) : 2019/ ISO 3838:2004	Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary stoppered pyknometer and graduated bicapillary pyknometer methods (*third revision*)
(Part 44) : 2013/ ISO 3839:1996	Determination of bromine number of distillates and aliphatic olefins — Electrometric method (*third revision*)
(Part 158) : 2016/ ISO 5661:1983	Hydrocarbon liquids — Determination of refractive index
8401 : 1994	Alkyl benzene sulphonic acid (acid slurry) — Specification (*first revision*)
7597 : 2001	Surface active agents — Glossary of terms (*first revision*)
13933 : 1995	Method of Test for Ready Biodegradability of Surface Active Agents (modified Sturm test)
ISO 12937 : 2000	Petroleum products — Determination of Water — Coulometric Karl Fischer Titration Method
ASTM D86-19	Standard Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure
ASTM D93-19	Standard Test Methods for Flash Point by Pensky — Martens Closed Cup Tester

IS No./International Standard	*Title*
ASTM D2710-09 (2018)	Standard Test Method for Bromine Index of Petroleum Hydrocarbons by Electrometric Titration
ASTM D4052-18a	Standard Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
ASTM D6304-16e1	Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fisher Titration
UOP304-08	Bromine Number and Bromine Index of Hydrocarbons by Potentiometric Titration
UOP481-10	Water in Liquid Hydrocarbons, and Heavy Hydrocarbons by Vaporizer, using Coulometry
UOP698-12	Linear Alkylbenzene (LAB) Product by GC

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 shall apply.

4 REQUIREMENTS

4.1 Description

Linear alkyl benzene shall be a clear, colourless liquid with a characteristic odour.

4.2 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in moist-free mild steel tanks or barrels or as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The container shall be securely closed and marked with the following particulars:

a) Name of the material;
b) Indication of source of manufacture;
c) Net mass of the material;
d) Batch number or lot number in code or otherwise; and
e) Month and year of manufacture.

5.2.2 For information on safety data of product, the material safety data sheet of manufacturer/supplier of product shall be referred.

5.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in IS 1447 (Part 1).

7 TESTS

7.1 Tests shall be carried out as prescribed in relevant standards and Annexes A to H of this standard. References to the relevant methods and Annexes A to H are given in col 4 of Table 1.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Linear Alkyl Benzene

(*Clauses* 4.2 *and* 7.1)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Colour		Annex A
	a) Saybolt, *Min*	30	
	or		
	b) In 5 inch cell on Lovibond scale, expressed as Y + 5R, *Max*	0.5	
ii)	Specific gravity at 15.5°/15.5°C	0.858-0.868	IS 1448 [Part 32] or ASTM D-4052
iii)	Moisture, ppm, *Max*	100	**5** of IS 286 or ISO 12937 or ASTM D-6304 or UOP 481
iv)	Doctor test	Negative	IS 1448 [Part 19]
v)	Flash point (Closed cup), °C, *Min*	130	IS 1448 [Part 21] or ASTM D-93
vi)	Bromine index, *Max*	10	UOP 304 or ASTM D 2710
vii)	Ready biodegradability of sulphonate [type test] (*see* Note)	To pass the test	IS 13933 or ASTM D 2667
viii)	Distillation, 95 percent recovery at °C, *Max*	320	IS 1448 [Part 18] or ASTM D-86
ix)	Molecular weight:		Annex B
	Low molecular weight	234-239	or
	High molecular weight	240-246	UOP 698-12
x)	Paraffin, percent by mass, *Max*	0.4	Annex C
xi)	Sulphonability, percent by mass, *Min*	98.0	Annex D
xii)	Chain-length distribution, percent:		
	a) Less than C10, *Max*	1.0	Annex E or UOP 698-12
	b) C10-C13	Shall not be less than 98	
	c) C14, *Max*	1.0	
xiii)	Refractive Index, at 20°C	1.48-1.49	Annex G or IS 1448 [Part 158]
xiv)	Tetralins (weight percent)	Less than 1.0	Annex H
xv)	LAB content, percent, *Min*	90	UOP 698-12

NOTES:

1 Linear alkyl benzene is to be sulphonated as given in Annex F before carrying out ready biodegradability test.

2 Sample shall be tested for this requirement every time whenever there is change in composition, source of raw material and process.

ANNEX A

(*Table* 1)

DETERMINATION OF COLOUR

A-1 GENERAL

The colour of the material can be determined by either Saybolt chromometer method or Lovibond tintometer method. However, in case of any dispute, Saybolt chromometer method shall be the referee method.

A-2 SAYBOLT CHROMOMETER METHOD

The colour shall be determined by the method specified in IS 1448 (Part 14).

A-3 LOVIBOND TINTOMETER METHOD

The colour shall be determined by the method prescribed in IS 1448 (Part 13).

ANNEX B

(*Table* 1)

DETERMINATION OF MOLECULAR WEIGHT AND CONCENTRATION OF LINEAR ALKYL BENZENE

B-1 OUTLINE

This method is used for the analysis of linear alkyl benzene and total nonlinear alkyl benzene. It is also used for the analysis of other side products in the sample. Sample is injected in a gas chromatograph and is passed through a capillary column coated with fused silica, where different components of sample adsorb on column substrate and eluted at different temperatures depending on molecular structure and molecular weight, along with the carrier gas. Flame ionization detector is used for the analysis.

B-2 APPARATUS

B-2.1 Chromatographic Fused Silica Column

B-2.2 Gas Chromatograph (able to control temperature of sample, injector port and column)

B-2.3 Data Processing System

B-2.4 Leak Detector Gas

B-2.5 Regulator for cylinders containing Air, Hydrogen and Nitrogen/Helium

NOTE — The desirable pressure range can be between (4-100 psi)

B-3 REAGENTS AND MATERIALS

B-3.1 Syringe (10 µl), for sample injection.

B-3.2 Auto sampler vials.

B-3.3 High purity carrier gas (zero gas) — Nitrogen or Helium with ultra-high purity of about 99.99 percent.

B-3.4 High purity hydrogen gas (zero gas) — 99.99 percent

B-3.5 Air (zero gas).

B-3.6 Gas purifier, in order to remove oxygen and moisture from gas.

B-3.7 Working standard solutions, of five different concentrations.

B-3.8 Standard stock solutions, for internal reference standards.

B-4 PROCEDURE

B-4.1 Set up the gas chromatograph as per the manufacturer’s instructions. Install fused silica capillary column and gas purifier (between cylinder and place where gas enters).

B-4.2 Set the column oven temperature to 250°C and maintain this temperature till a stable base line is achieved.

B-4.3 Now, lower down the column temperature to about 100°C.

B-4.4 Inject the standard solution along with internal reference standard and start the column program.

B-4.5 Concentration of linear alkyl benzene can be calculated from the peak area of each isomer.

B-4.6 Repeat the process given in **B-4.4** for each standard solution (*See* **B-3.7**).

B-5 CALCULATIONS

B-5.1 The percentage of individual Linear alkyl benzene chain is calculated as follows:

$$\text{Mass percent} = 100 \times \frac{W}{S}$$

where

W = peak area for linear alkyl benzene.

S = sum of the recorded peak area for linear alkyl benzene and side products formed during the process.

100 = factor to convert to mass percent.

B-5.2 Average molecular weight of linear alkyl benzene in the sample $= \dfrac{100}{\sum_{i=1}^{n} \dfrac{M_i}{G_i}}$

Where,

G_i = molecular weight of component i, g/mol,

M_i = mass percentage of component i,

n = number of each type of components formed, and

100 = in order to convert from percentage mass.

B-6 PROPOSED OPERATIONAL CONDITIONS

Temperature of Injection port	**250°C**
Column Temperature	
Initial time	2 min
Programming rate (A)	2°C/min
Initial temperature	100°C
Intermediate temperature	180°C
Programming rate (B)	10°C/min
Final temperature	250°C
Detector	**Flame Ionization Detector**
Hydrogen flow rate	30 ml/min
Air flow rate	350 ml/min
Sample Size	
Fractioned sample	0.2 µl
Non fractioned sample	1.0 µl
Carrier gas	Helium or Nitrogen
Flow rate	1.4 ml/min
Column Dimensions	30m × 0.25 mm × 0.25 µm

ANNEX C

(*Table* 1)

DETERMINATION OF PARAFFINS

C-1 OUTLINE OF THE METHOD

The sample to be analysed is injected into a gas chromatograph that is equipped with fused silica capillary column. A quantitative blend, containing known amount of normal paraffins in linear alkyl benzene (LAB) is chromatographed under identical conditions. The calibration chromatogram thus developed is used to ascertain the normal paraffins in the sample by comparing the retention of the normal paraffins to the observed retention times of the components obtained in the synthetic mixture. Quantitative results are obtained from the measured areas of the recorded peaks, adjusted for differences in response by the use of factors determined from the analysis of synthetic blend.

C-2 PROCEDURE

C-2.1 Inject C_{10} to C_{14} individual normal paraffins namely decane, undecane, dodecane, tridecane and tetradecane into gas chromatograph and find out the retention times of each paraffin. Prepare a synthetic blend containing close to 0.2 by mass of total normal paraffin in linear alkyl benzene. Now inject the synthetic blend and run the chromatogram. Identify the normal paraffin peaks from C_{10} to C_{14}. Calculate the area percent of individual normal paraffin and sum it up to get total area percent of the total normal paraffin in the blend. Keep the column operational conditions same as given in **B-6**. Calculate the area response factor as given in **C-3.1.**

C-2.2 Inject linear alkyl benzene sample and run the chromatogram. From the area percent of total normal paraffin, calculate the weight percent as given in **C-3.2**.

C-3 CALCULATION

C-3.1 Area response factor, $F = \frac{W}{A}$

where

W = weight percent of total normal paraffin in the blend; and

A = area percent of total normal paraffin in the blend.

C-3.2 The weight percent of total normal paraffin in linear alkyl benzene = $B \times F$

where

B = area percent of total normal paraffin obtained from the chromatogram; and

F = response factor of total normal paraffin.

ANNEX D

(*Table* 1)

DETERMINATION OF SULPHONABILITY

D-1 OUTLINE OF THE METHOD

A known mass of linear alkyl benzene of known molecular mass is sulphonated with 20 percent oleum. Knowing the mass of the product after sulphonation and its active matter content, the percent sulphonability is calculated.

D-2 APPARATUS

A four-necked round bottom flask having 500 ml capacity, with standard joints, is fitted with a mercury-sealed stirrer, thermometer (0-110°C) and a 100-ml, capacity addition funnel with pressure equalising side-arm (*see* Fig. 1). A guard-tube filled with granular, fused calcium chloride is inserted into the fourth neck of the flask.

D-3 REAGENTS

D-3.1 20 Percent Oleum

D-3.2 Purified Hyamine 1622

Commercially available Hyamine 1622 is crystallised 6 to 7 times from chloroform-petroleum ether (60-80°). In the last crystallisation, it is dissolved in chloroform and filtered through filter paper to remove traces of dust and dirt. Then, the chloroform solution is concentrated on a hot-water bath and petroleum ether is added to the hot solution until it is slightly hazy. This solution is then cooled, scratching the sides with a clean glass rod. The separated crystals are filtered in a Buchner funnel under vacuum and the cake of crystals pressed dry with an inverted bottle stopper. The crystals are then scraped gently from the filter-paper into a glass dish and dried subsequently on a steam-bath. The melting point of the dried crystals is 163.5°C (Literature, 164-166°C).

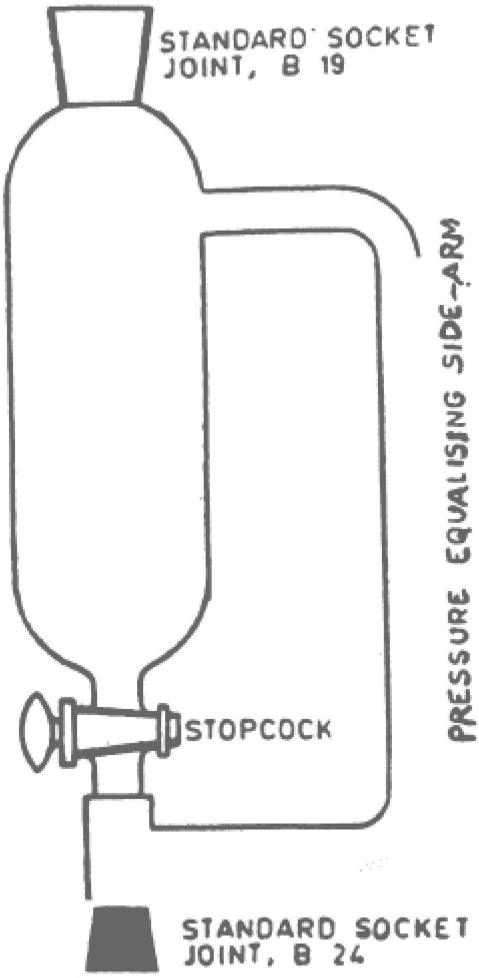
STANDARD SOCKET JOINT, B 19
PRESSURE EQUALISING SIDE-ARM
STOPCOCK
STANDARD SOCKET JOINT, B 24

FIG. 1 APPARATUS FOR DETERMINATION OF SULPHONABILITY

D-4 PROCEDURE

The empty 500-ml round bottomed flask, fitted with stirrer and thermometer (partly coming in contact with the reaction mixture) is weighed to the second decimal. 100.00 g of the linear alkyl benzene sample is now weighed into it. The mercury seal and calcium chloride guard tube are fitted. 120 g oleum (*see* **D-3.1**) is weighed into the addition funnel, which is stoppered at once and fitted to the 4-necked flask. The linear alkyl benzene is then cooled under stirring in a bath containing ice and water to 20°C. Dropwise addition of the 20 percent oleum is started. The reaction is exothermic, but, by adding more ice to the bath, reaction temperature is held below 25°C and a fairly fast rate of addition of oleum is maintained. After complete addition of oleum, reaction mixture temparature is allowed to come up to 40°C by replacing ice and water in the bath with warm water. The reaction mixture is maintained at 40°C with stirring for 1 h. The calcium chloride guard tube, additional funnel and mercury seal are removed. The outside of the flask is wiped dry and weighed, as before, with stirrer and thermometer, to the second decimal. The net weight of the contents after sulphonation (M_1) is thus determined. Its anionic active matter content is then determined as given in **A-4.2** of IS 8401 using hyamine 1622 solution.

D-5 CALCULATION

$$\text{Percent sulphonability} = \frac{M_1 \times A \times M_2}{100 \times (M_2 + 80)}$$

where

M_1 = nett mass of reaction mixture after sulphonation;

A = percent active matter of the sulphonated mixture; and

M_2 = average molecular mass of the sample.

ANNEX E

(*Table* 1)

DETERMINATION OF CHAIN LENGTH DISTRIBUTION

The gas liquid chromatogram of the sample is taken and recorded as described in **C-2** and assignments made as in **B-4**. Thereafter, the chain length distribution of the sample is calculated as given in **B-5.1.**

ANNEX F

(*Note* under *Table* 1)

METHOD FOR SULPHONATION OF LINEAR ALKYL BENZENE

Take 240 g of linear alkyl benzene into a three-neck flask having a capacity of 1 l. Add 240 g of 15-20 percent oleum to it slowly from the funnel attached to the flask, under constant stirring. Maintain the temperature of the mass between 30°C and 35°C by cooling the flask in a water bath. After the addition of oleum is over, continue stirring for a further period of three hours maintaining the temperature of the water bath at 45-50°C. Add 40 ml water and stir thoroughly. Transfer the material to a separating funnel and allow it to separate into two layers. The separation takes 4-5 h. Discard the lower layer of spent acid. Take the upper layer, which is sulphonated for the ready biodegradability analysis.

ANNEX G

(*Table* 1)

DETERMINATION OF REFRACTIVE INDEX

G-1 PRINCIPLE

Refractive index is the ratio of the velocity of light in air to its velocity in the substance. The value of refractive index of liquids varies inversely with both wavelength and temperature. It is the ratio of sine of angle of incidence and sine of angle of refraction when light passes from air to substance.

G-2 APPARATUS

G-2.1 High Resolution Refractometer — Optical-Mechanical Refractometer of Abbe type.

G-2.2 Temperature Control Unit — Equipped with pump for maintaining the prism temperature within 0.1°C. There should be an external liquid bath with both heating and cooling capability. Using external liquid bath, the liquid might pass the device for measurement while leaving the prism and not while entering the prism. The temperature control unit should have a stability and uniformity of ±0.1°C each.

G-2.3 Temperature Measuring Device

Either of the following may be used —

G-2.3.1 Apparatus using mercury in glass thermometer having a range of 19-27°C.

G-2.3.2 Apparatus employing platinum resistance probe, thermocouple or equivalent temperature sensor.

G-2.4 Light Sources

In conjunction with optical mechanical refractometer, these light sources shall be used-

G-2.4.1 *Sodium Arc lamp* **—** Na_D line at 589 nm

G-2.4.2 *Mercury Arc lamp* **—** Hg_c line at 546 nm or Hg_g line at 436 nm.

G-2.4.3 *Helium Discharge lamp*

G-2.4.4 *Mercury Cadmium lamp*

G-2.4.5 *Cadmium Arc lamp*

G-2.5 Light Filters

Multiple borderlines are observed using Arc lamps; therefore use of filters is necessary. Choice of filter is generally based on instrument design and as recommended by the manufacturer.

G-3 REAGENTS AND MATERIALS

G-3.1 N-pentane, 95 mol percent purity, *min* **—** used for cleaning faces of prisms (Warning — It is flammable and harmful if inhaled).

G-3.2 Toluene (HPLC Grade) **–** used for cleaning faces of prism.

G-3.3 1-Bromonapthalene (Purity of 98 percent, *min*) — used as contact liquid when calibrating with reference standard.

G-3.4 Primary Reference Materials

G-3.4.1 Solid reference standards with refractive index value engraved

G-3.4.2 Distilled or deionized water

G-3.4.3 2,2,4-trimethylpentane (99 percent purity, *min*)

G-3.4.4 Methylcyclohexane (99 percent purity, *min*)

G-3.4.5 Toluene (99 percent purity, *min*).

G-3.5 Secondary reference materials, for calibrating the instrument.

G-3.5.1 *Mineral oil calibration standards*, certified for specific refractive index ranges and temperatures.

G-4 PROCEDURE

G-4.1 Sampling

A sample of at least 0.5 ml is required. There shall be no water, suspended solids or other materials in the sample. They may settle on the surface of the prism and affect the reading. An hydrous Calcium chloride can be used to remove water, followed by filtering or centrifuging in order to remove the desiccant.

G-4.2 Procedure A

G-4.2.1 *Apparatus preparation*

Refractometer should be clean at all the times as oil and dust may impair the optical component of the instrument. First, clean faces of prism with toluene and then with n-pentane using cotton swab or any other material as per manufacturer’s instructions. Do not use dry cotton to rub faces of the prism.

Adjust the temperature control system such that the temperature measuring device is 0.1°C of the desired value. Leave the refractometer to equilibrate for 30 min after turning on the light source.

G-4.2.2 *Calibrating Refractometer using solid reference standard*

Clean the prism faces and surface of solid reference standard. Apply a drop of 1-bromonapthalene liquid to the centre of the polished surface of solid reference material. Press the solid reference standard against the surface of prism, keeping its polished end towards the source of light.

In order to calibrate the instrument using solid reference standard, manufacturer's instructions shall be followed.

If the value of refractive index observed differs from the engraved value by 0.000 1, adjust the refractometer's scale reading as per the instructions of manufacturer.

G-4.2.3 *Calibrating the refractometer using liquid reference standards*

As given in **G-4.2.4**, adjust the refractive index of the material. Reading should not differ by more than 0.000 1 of the given value. Adjustment can be made as per manufacturer's instructions.

G-4.2.4 *Method*

G-4.2.4.1 Ensure that the prism faces are clean and dry. The temperature of the prism shall be within ± 0.1°C of the desired temperature.

G-4.2.4.2 Open the prism assembly and place the drop of the sample on the lower face of the prism. Lock and close the prism assembly. Turn on the light. Allow 3 to 4 min for temperature equilibration.

G-4.2.4.3 Observe light and dark portions from the eye piece. Adjust the instrument so that the boundary between light and dark portions is sufficiently sharp. Adjust the instrument till the sharp boundary line intersects the mid-point of the cross hairs.

G-4.2.4.4 Determine the value of refractive index from the scale. Take 3 to 4 readings and calculate the average.

G-4.2.4.5 If the instrument is capable of measuring refractive dispersion, change the source of light to one having a different wavelength and determine refractive index as prescribed in **G-4.2.4.3** and **G-4.2.4.4**.

G-4.2.5 *Calculations*

G-4.2.5.1 Report the average refractive index upto four decimal places. Also, report the temperature at which the readings were made.

Example: η_D = x.xxxx at 20°C Or η_{589} = x.xxxx at 20°C

NOTE — D or 589 indicates that Sodium D spectral line was used for the reading.

G-4.2.5.2 If the measurement of refractive dispersion was made, calculate the refractive index value at two different wavelengths and multiply their difference by 10 000.

Example: $(\eta_g - \eta_D) \times 10^4$ at 20°C, where g indicates Mercury and D indicates Sodium.

G-4.3 Procedure B-Automatic Procedure

G- 4.3.1 *Apparatus*

G-4.3.1.1 *Automatic digital refractometer*, with a measuring range between 1.330 0 to 1.500 0 or higher may be used. The instrument shall incorporate a solvent resistant prism seal.

G-4.3.1.2 *Temperature control unit — see* **G-2.2**.

G-4.3.1.3 *Temperature measuring device* — A platinum resistance probe having an accuracy and resolution of at least 0.1°C. An equivalent temperature sensor may also be used.

G-4.3.1.4 *Light source* — White light, for example, obtained using a tungsten halogen lamp or light emitting diode, and is able to provide sodium D spectral line at a wavelength of 589 nm.

G-4.3.1.5 *Light Filters — see* **G-2.5**.

G-4.3.2 *Reagents and materials* — as prescribed in **G-3**.

G-4.3.3 *Apparatus* — as prescribed in **G-4.2.1**.

NOTE — It should be ensured that the instrument has a solvent resistant prism seal.

G-4.3.4 *Calibration of the refractometer*

Using liquid reference standards, automatic refractometers shall be calibrated following manufacturer's instructions.

G-4.3.5 *Procedure*

G-4.3.5.1 Make sure that the prism and surrounding plate is cleaned.

G-4.3.5.2 Make sure that the prism temperature is stabilized within ±0.1°C. Calibrate the instrument properly.

G-4.3.5.3 Introduce the test sample to the prism after ensuring that the prism is properly covered. In order to allow thermal equilibration, do not introduce excess amount of sample.

G-4.3.5.4 Take multiple readings till steady value is obtained.

G-4.3.6 *Results*

Direct and digital reading of refractive index is given by the instrument. The readings can be recorded manually or electronically.

Report the average refractive index upto four decimal places. Also, report the temperature at which the readings were made.

Example: η_D= x.xxxx at 20°C Or η_{589} = x.xxxx at 20°C

NOTE — D or 589 indicates that Sodium D spectral line was used for the reading.

ANNEX H

(*Table* 1)

DETERMINATION OF TETRALINS

H-1 PRINCIPLE

Tetralins are analyzed using isocratic High Performance Liquid Chromatography (HPLC) technique and ultraviolet detector. 2, 2, 4-trimethyl pentane is used as mobile phase and microparticulate silica is used as a stationary phase. The sample is quantified using external standard of tetrahydronapthalene. Tetralins are not available commercially so it is assumed that commercially available tetrahydronapthalene gives similar molar response as the tetralins.

H-2 REAGENTS

During analysis, it is preferred that reagents are of analytical grade or HPLC grade and do not interfere with the results.

H-2.1 Reagent Grade Water

H-2.2 2,2,4-trimethyl pentane

H-2.3 Tetrahydronapthalene

H-2.4 Hexane

H-2.5 Toluene

H-3 APPARATUS

High performance liquid chromatograph instrument with chromatographic column of normal phase which is 250 mm long with an internal diameter of 4 mm containing micro particulate silica.

H-4 PROCEDURE

H-4.1 Preliminary Test

H-4.1.1 Length of the column (one or two columns in series) and the flow rate should be arranged and optimised properly in order to increase the efficiency and resolution of the column. One of the methods is to run a standard solution of toluene and tetrahydronapthalene in hexane. Washing of the column with polar solvent is not recommended.

H-4.1.2 Determine net retention time (t'), capacity factor for both the solvents (Toluene and THN). Calculate the separation factor as the ratio of the capacity factors of the solvents.

H-4.1.3 A typical example is given below:

Reagent	*Net Retention Time*, $t' = t - t_0$ (min)	*Capacity Factor*, $K' = t'/t_0$
Toluene	4.8	0.94
THN	6.3	1.24

NOTE — t_o is elution time at the dead volume. The elution time is 5.1 min

$$\text{Separation factor} = \frac{K'_{\text{THN}}}{K'_{\text{Toluene}}} = 1.32$$

For an accurate analysis, ensure that the separation factor is greater than or equal to 1.20.

H-4.2 Calibration

H-4.2.1 Prepare a solution of 0.1 g/l of tetrahydronapthalene. For this, weigh 100 mg of the tetrahydronapthalene to the nearest 0.1 mg. Use 1 l volumetric flask to make up the volume using 2,2,4-trimethylpentane. Dilute, if required.

H-4.2.2 Operating instructions for HPLC —

Column	
Material	Stainless steel
length × internal diameter	250 mm × 4 mm
Packing	microparticulate silica, 5 µm

Mobile Phase	
Solvent	2,2,4-trimethylpentane
Flow rate	0.5 ml/min - 1 column
	1.0 ml/min - 2 columns
Programme	Isocratic for 30 min

Detector System

Detector System	
Type	Ultraviolet absorbance
Wavelength	254 nm
Temperature	Room temperature
Injection volume	20 µl

In case the mean molar mass is unknown, analyse the sample and find carbon number using gas chromatography peak area normalization technique.

Calculate the average molar mass of the sample as -

$$M_T = \frac{100}{\sum_{i=8}^{15} \frac{W_{LAB,i}}{M_i}}$$

where

$W_{LAB,i}$ = mass percentage of linear alkyl benzene at carbon number 'i' of alkyl chain.

M_i = molar mass of tetralins at carbon number 'i' of alkyl chain, g/mol.

H-4.3 Calculation

Determine the content of tetralins, W_T, expressed as mass fraction in percent, in the sample as follows:

$$W_T = \frac{A_S}{A_{THN}} \times \frac{THN(c)}{S} \times \frac{100}{R}$$

where

A_s = area under the peak for tetralins obtained by analysing the filtered solution by HPLC.

$THN(c)$ = mass concentration of THN in the calibration solution, g/l.

R = molar mass ratio = $\frac{M_{THN}}{M_T} = \frac{132}{M_T}$

A_{THN} = Area under the peak for THN obtained by analysing the solution using HPLC.

S = mass concentration of sample, g/l.

ANNEX J

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

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